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Indian Standard

SPECIFICATION FOR DEXTRIN FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

UDC 664·161 : 662·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

July 1988

Indian Standard

SPECIFICATION FOR DEXTRIN FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 29 January 1988, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 The Bureau has already published IS : 6367 Dextrin for adhesive industry, IS: 4269 Dextrin for foundries, and IS : 5448 Dextrin for textile industry. The present standard is prepared to cater to explosive and pyrotechnic industry, the requirements for which are different from the above mentioned fields.

0.3 Dextrins are the degradation products of starch generally obtained from potato, corn or topioca. In explosive industry, the dextrins are mainly used as crystal modifier and in the pyrotechnic industry, they are used as core binders.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for dextrin for use in explosive and pyrotechnic industry.

2. TYPES

2.1 The dextrin shall be of two types:

Type 1 — for use in explosive industry, and

Type 2 — for use in pyrotechnic industry.

3. REQUIREMENTS

3.1 The meterial shall consist essentially of dextrin in the form of amorphous fine powder of white or light yellow colour. It shall be free from grit and visible impurities.

3.2 The material shall have particle size as agreed to between the purchaser and the supplier.

3.3 The material shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Appendix A.

3.4 The material shall conform to the requirements of this standard for a minimum period of one year from the date of manufacture under normal conditions of storage.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in polyethylene bags duly sealed and then inserted in metallic or alkathene drums or as agreed to between the purchaser and the supplier. The containers shall be stencilled suitably.

TABLE 1 REQUIREMENTS FOR DEXTRIN FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

(*Clause* 3.3)

Sl No.	Characteristic	Requirement		Method of Test, Ref to
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, *Max*	10·0	10·0	A-2
ii)	Solubility, percent by mass			
	a) in cold water	22-40	60 (*Min*)	A-3
	b) in hot water, *Min*	95	95	A-4
iii)	Ash content, percent by mass, *Max*	0·5	0·5	A-5
iv)	Solid impurity (sand), percent by mass, *Max*	0·005	0·05	A-6
v)	Acidity (as H_2SO_4), *Max*	0·10	3·2-3·4	A-7
vi)	Reducing sugar (as dextrose), percent by mass, *Max*	5·0	5·0	A-8
vii)	Water soluble sulphate, percent by mass, *Max*	0·3	—	A-9
viii)	Water soluble chlorides, percent by mass, *Max*	0·15	—	A-10
ix)	Retrogradation test*	(Shall pass the test)		A-11
x)	Practical test*	(Shall pass the test)		A-12

*If agreed to between the purchaser and the supplier.

4.2 Marking — Each package shall be legibly and indelibly marked with the following information:

a) Name of the material;

b) Net mass of the material;

c) Manufacturer's name and/or his trade-mark, if any;

d) Month and year of manufacture; and

e) Lot number or batch number in code or otherwise.

4.2.1 The packages may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.3)

METHODS OF TEST FOR DEXTRIN FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MOISTURE CONTENT

A-2.1 **Procedure** — Weigh accurately about 3 to 5g of the sample into a weighing bottle which has been dried, cooled in the desiccator and then weighed. Place the weighing bottle in an air/water oven after removing the lid at 100 ± 5°C till constant mass is obtained. Remove the weighing bottle from the oven, cover the lid and cool in a desiccator to room temperature and weigh. Repeat this procedure. Keep the weighing bottle in the oven only for half an hour each time until the difference between two successive weighings is less than 1 mg.

A-2.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = loss in mass in g of the sample upon drying, and

M = mass of the sample taken for the test.

A-3. DETERMINATION OF SOLUBILITY IN COLD WATER

A-3.1 Weigh accurately 10 g of the sample, the moisture of which has been previously determined, into a 250-ml beaker. To the sample add a quantity of water calculated to give an anhydrous content of 7·9 percent (*m/m*). This volume of water is calculated as given in A-3.2. Stir the mixture and allow it to stand for one hour at room temperature, stirring occasionally. The total volume is then measured (*see* Note 1), and the suspension centrifuged for 10 minutes at 3 000 rpm. Transfer 20 ml of the clear supernatant liquor by means of a pipette to a tared flat-bottomed aluminium dish. Evaporate the liquor to dryness in an oven at 105°C (*see* Note 2).

Calculate the increase in mass of the aluminium dish to a percentage on the mass of dry sample taken and report the result as percentage soluble in water.

NOTE 1 — 10 g of dextrin, added to a known volume of water, has been found to increase the volume by approximately 7·5 ml. For routine test purposes, the total volume of water and dextrin can be taken as $V + 7{\cdot}5$ ml.

NOTE 2 — Usually 4-5 hours is sufficient to dry the samples, but they can be left drying overnight, with little or no detrimental effect. Care must be taken to cover the dried residue, as it is very hygroscopic. A watch glass should be used to cover the residue, and the dish is cooled in a desiccator.

A-3.2 Calculation

Volume of water in ml required to give an anhydrous content of 7·9 percent:

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*Specification for water for general laboratory use (*second revision*).

$$V = \frac{\left(10 - \frac{m}{10}\right) \times 100 - 10}{7 \cdot 9}$$

where m is the moisture content of the sample in percent.

A-4. DETERMINATION OF SOLUBILITY IN HOT WATER

A-4.1 Procedure — Weigh 25 g of dextrin and put it into a porcelain mortar, add 100 ml distilled water and rub the dextrin until all clots disappear. Transfer into a beaker, warm to 60-80°C with constant stirring for complete dissolution. Transfer to a 250-ml flask, cool and make up to mark.

Pipette out an aliquot of 25 ml and evaporate to dryness in pre-weighed glass bowl/dish. Note the weight of residue as R.

$$\text{Solubility, percent by mass} = \frac{R \times 100}{2 \cdot 5 \left(1 - \frac{M}{100}\right)}$$

where M is the moisture volume.

A-5. DETERMINATION OF ASH

A-5.1 Procedure — Weigh accurately about 10 g of the sample into a crucible. Incinerate on a low flame to prevent the material from swelling. Introduce in the muffle furnace for about half an hour at 800°C. Cool the crucible with the ash in a desiccator and weigh. Calculate the ash content of the dextrin on dry basis. The duplicate tests should not differ by more than 0·3 percent.

Ash, percent by mass, on dry basis

$$= \frac{(G_1 - G_0)}{G_2 - G_0} \times \frac{100}{(100 - M)} \times 100$$

where

G_1 = mass of crucible with ash in g,

G_0 = mass of crucible in g,

G_2 = mass of crucible and the sample in g, and

M = moisture content of the dextrin percent.

A-6. DETERMINATION OF SOLID IMPURITY (SAND)

A-6.1 Procedure — Weigh 50 g of dextrin on an analytical balance and transfer to a 1 000-ml beaker. Add 500 ml of distilled water and 100 ml of 25 percent hydrochloric acid. Stir the contents of the beaker till the dextrin is dissolved completely. Allow to cool and settle for 10 to 15 minutes. Decant the sediment and allow it to rest, and pour off the supernatant liquid. Repeat the decantation for 3 to 4 times, before transfer to a filter paper. Wash the residue. Place the filter paper with the residue into a pre-weighed crucible and dry in a muffle to a constant mass.

Calculate the amount of sand percent on dry basis of the sample.

$$\text{Sand, percent by mass} = \frac{G_1 - G_0 \times 2 \times 100}{100 - W}$$

where

G_1 = mass of crucible and sand in g,

G_0 = mass of the empty crucible in g,

W = moisture in the dextrin percent, and

$100 - W$ = dry substance of the dextrin percent.

A-7. DETERMINATION OF ACIDITY

A-7.1 Procedure — Dissolve 5 g of the prepared sample in 250 ml distilled water and, if necessary, filter the solution. Boil the clear liquid for two minutes, then cool and titrate it with 0·1 N sodium hydroxide, using methyl red as indicator. From the volume of sodium hydroxide solution required, calculate acidity as a percentage of sulphuric acid on the mass of sample taken and report the result as percent sulphuric acid (H_2SO_4)

1 ml 0·1 N NaOH = 0·004 9 g H_2SO_4

A-8. DETERMINATION OF REDUCING SUGARS

A-8.1 Reagents

A-8.1.1 *Lead Subacetate Solution* — Activate about 200 g of litharge by heating at 650°C for 3 h (cooled product should be of lemon colour). Boil 430 g of neutral lead acetate, 130 g of freshly activated litharge and one litre of water for 30 min. Cool and allow to settle. Dilute the supernatant liquid to density 1·25 with freshly boiled and cooled water.

A-8.1.2 *Potassium Oxalate* — crystalline.

A-8.1.3 *Fehling's Solution (Soxhlet Modification)* — prepared by mixing immediately before use, equal volumes of Solution A (**A-8.1.3.1**) and Solution B (**A-8.1.3.2**).

A-8.1.3.1 *Solution A* — Dissolve 34·639 g of copper sulphate ($CuSO_4 . 5H_2O$) in water, add 0·5 ml of concentrated sulphuric acid (analytical reagent), and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.

A-8.1.3.2 *Solution B* — Dissolve 173 g of Rochalle salt (potassium sodium tartrate, $KNaC_4H_4O_6 . 4H_2O$) and 50 g of sodium hydroxide, analytical reagent in water, dilute to 500 ml in a volumetric flask and allow the solution to stand for two days. Filter this solution through prepared asbestos.

A-8.1.4 *Methylene Blue Indicator Solution* — Dissolve 0·2 g of methylene blue in water and dilute to 100 ml.

A-8.1.5 *Stock Solution of Dextrose* — Weigh accurately about 10 g of anhydrous dextrose into a 1-litre volumetric flask and dissolve it in water. Add to this solution, 2·5 g of benzoic acid, shake to dissolve the benzoic acid and make up the volume to the mark with water. This solution should not be used after 48 hours.

A-8.1.6 *Standard Dextrose Solution* — Dilute a known aliquot of the stock solution of dextrose (**A-8.1.5**) with water containing 0·25 percent (*m/v*) benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it shall be required to reduce all the copper in the Fehling's solution taken for titration in **A-8.2**. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml. Prepare this solution fresh every day.

NOTE — When 10 ml of Fehling's solution (*see also* Note 2 under **A-8.2**) are taken for titration, a standard dextrose solution containing 0·11 to 0·30 percent (*m/v*) of anhydrous dextrose is convenient for use. With 25 ml of Fehling's solution, a standard dextrose solution containing 0·25 to 0·75 percent of anhydrous dextrose should be used.

TABLE 2 DEXTROSE FACTORS FOR 10 ml OF FEHLING'S SOLUTION

TITRE	*DEXTROSE FACTOR	ANHYDROUS DEXTROSE PER 100 ml OF SOLUTION
(1)	(2)	(3)
15	49·1	327
16	49·2	307
17	49·3	289
18	49·3	274
19	49·4	260
20	49·5	247·4
21	49·5	235·8
22	49·5	225·5
23	49·7	216·1
24	49·8	207·4
25	49·8	199·3
26	49·9	191·8
27	49·9	184·9
28	50·0	178·5
29	50·0	172·5
30	50·1	167·0
31	50·2	161·8
32	50·2	156·9
33	50·3	152·4
34	50·3	148·0
35	50·4	143·9
36	50·4	140·0
37	50·5	136·4
38	50·5	132·9
39	50·6	129·6
40	50·6	126·5
41	50·7	123·6
42	50·7	120·8
43	50·8	118·1
44	50·8	115·5
45	50·9	113·0
46	50·9	110·6
47	51·0	108·4
48	51·0	106·2
49	51·0	104·1
50	51·1	102·2

*Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling's solution.

A-8.2 Standardization of Fehling's Solution — Pour standard dextrose solution (*see* **A-8.1.6**) into a 50-ml burette (*see* Note 1). Find from Table 2 the titre (volume of standard dextrose solution required to reduce all the copper in the 10 ml of Fehling's solution) corresponding to the concentration of standard dextrose solution. (For example, if the standard dextrose solution contains 167·0 mg of anhydrous dextrose per 100 ml, the corresponding titre would be 30 ml). Pipette 10 ml (*see* Note 2) of Fehling's solution into a 300 ml conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper so that not more than 1 ml will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently, boil the contents of the flask for 2 minutes. At the end of 2 minutes of boiling, add, without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears.

[The titration should be completed within 1 minute so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 3).] Note the titre in millilitres of standard dextrose solution. Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in 1 ml of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 2 and determine correction, if any, to be applied to the dextrose factors derived from Table 2.

NOTE 1 — In adding sugar solution to the reaction mixture, the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of the steam while adding sugar solution. Burettes with glass taps are unsuitable for this work, as the taps become heated by the steam and are liable to jam.

NOTE 2 — Instead of using 10 ml of Fehling's solution, a 25 ml portion may be used for standardization of dextrose solution and also for titration of test solution (**A-8.3.1.2**). In that case, Table 3 shall be used in place of Table 2.

NOTE 3 — It should be observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

Example

Concentration of anhydrous dextrose in standard dextrose solution, mg/100 ml = 167·0

Titre obtained by direct titration = 30·1 ml

Dextrose factor for 30·1 ml of standard dextrose solution = 30·1 × 1·670 = 50·267 0

TABLE 3 DEXTROSE FACTORS FOR 25 ml OF FEHLING'S SOLUTION

[*Clauses* A-8.2 (*Note* 2) *and* A-8.4]

TITRE	*DEXTROSE FACTOR	ANHYDROUS DEXTROSE PER 100 ml OF SOLUTION
(1)	(2)	(3)
15	120.2	801
16	120.2	751
17	120·2	707
18	120·2	668
19	120·3	633
20	120·3	601·5
21	120·3	572·9
22	120·4	547·3
23	120·4	523·6
24	120·5	501·9
25	120·5	482·0
26	120·6	463·7
27	120·6	446·8
28	120·7	431·1
29	120·7	416·4
30	120·8	402·7
31	120·8	389·7
32	120·8	377·6
33	120·9	366·3
34	120·9	355·6
35	121·0	345·6
36	121·0	336·3
37	121·1	327·4
38	121·2	318·8
39	121·2	310·7
40	121·2	303·1
41	121·3	295·9
42	121·4	289·0
43	121·4	282·4
44	121·5	276·1
45	121·5	270·1
46	121·6	264·3
47	121·6	258·8
48	121·7	253·5
49	121·7	248·4
50	121·8	243·6

*Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling's solution.

(titre in millilitre × number of milligrams of anhydrous dextrose in ml of standard dextrose solution)

Dextrose factor for 30·1 ml of standard dextrose solution from Table 2 (calculated by interpolation) = 50·11

Correction to be applied to the dextrose factors derived from Table 2 = 50·2670 − 50·11 = + 0·1570

A-8.3 Procedure — Weigh accurately about 10 g of the test sample in a beaker. Mix it thoroughly with 100 ml of water. Transfer to a 250-ml volumetric flask. Add drop by drop lead subacetate solution till precipitation is complete. Make the solution up to the mark by adding water. Mix thoroughly and allow it to settle down. Filter the supernatant liquid and collect the filtrate in a dry beaker. Add dry potassium oxalate to the filtrate to remove excess of lead. Filter and collect the filtrate (as a clarified solution) in a 250-ml flask. Make it up to the mark for subsequent estimations of reducing sugar (**A-8.3.1**).

A-8.3.1 *Estimation of Reducing Sugars*

A-8.3.1.1 *Incremental method of titration* — Pour the clarified solution (**A-8.3**) into a 50-ml burette (*see* Note 1 under **A-8.2**). Pipette 10 ml of Fehling's solution (*see* Note 2 under **A-8.2**) into a 300 ml conical flask and run in from the burette 15 ml of the clarified solution. Without further dilution, heat the contents of the flask over a wire gauze and boil. After the liquid has been boiling for about 15 s, it will be possible to judge if the copper is almost reduced by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide. When it is judged that nearly all the copper is reduced, add 1 ml of methylene blue indicator solution (*see* Note 1). Continue boiling the contents of the flask for 1 to 2 minutes from the commencement of ebullition, and then add the clarified solution in small quantities (1 ml or less at a time) allowing the liquid to boil for about 10 s between successive additions, till the blue colour of the indicator just disappears (*see* Note 3 under **A-8.2**). In case there appears to be still much unreduced copper, after the mixture of Fehling's solution with 15 ml of the clarified solution has been boiling for 15 s, add the clarified solution from the burette in large increments (more than 1 ml at a time, according to judgement) and allow the mixture to boil for 15 s after each addition. Repeat the addition of the clarified solution at intervals of 15 s until it is considered unsafe to add a large increment of the clarified solution. At this stage, continue boiling for an additional 1 to 2 min, add 1 ml of methylene blue indicator solution and complete the titration by adding the clarified solution in small quantities (less than 1 ml at a time) (*see also* Note 2).

NOTE 1 — It is advisable not to add the indicator until the neighbourhood of the end point has been reached, because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

NOTE 2 — When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration, but for the utmost degree of accuracy of which the method is capable, a second titration should be carried out by the standard method of titration (*see* **A-8.3.1.2**).

A-8.3.1.2 *Standard method of titration* — Pipette 10 ml of Fehling's solution (*see* Note 2 under **A-8.2**) into a 300-ml conical flask and run in from the burette almost the whole of the clarified solution required to effect reduction of all the copper (determined under **A-8.3.1.1**) so that, if possible, not more than 1 ml shall be required later to complete the titration. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add, without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add the clarified solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. The titration should be completed within 1 min so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 3 under **A-8.2**).

NOTE — The indicator is so sensitive that it is possible to determine the end point within one drop of the clarified solution in many cases. The complete decolourization of the methylene blue is usually indicated by the whole reaction liquid in which the cuprous oxide is continuously churned up becoming bright red or orange in colour. In case of doubt, the flame may be removed from the wire gauze for 1 or 2 s and the flask held against a sheet of white paper. (A holder of paper, suitably fixed round the neck of the flask, is very convenient for this purpose as it can be left round the neck of the flask, without risk of overbalancing it). The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is advisable not to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access to flask but there is no danger of this as long as continuous stream of steam is issuing from the mouth of the flask.

A-8.4 Calculation — Refer to Table 2 (or Table 3 as appropriate) for the dextrose factor corresponding to the titre (determined as given under **A-8.3.1.2**) and apply the correction previously determined under **A-8.2**. Calculate the dextrose content of the clarified solution (*see* **A-8.3**) as follows:

$$\text{Anhydrous dextrose present in 1 ml of the clarified solution, mg} = \frac{\text{dextrose factor}}{\text{titre}}$$

NOTE — Tables 2 and 3 show, for the standard method of titration, the values corresponding to integral millilitres of the sugar solutions; values corresponding to intermediate figures shall be obtained by interpolation.

A-8.4.1 Reducing sugars, percent by mass (on dry basis)

$$= \frac{mV}{10\,M} \times \frac{100}{100 - A}$$

where

m = anhydrous dextrose in mg present in 1 ml of clarified solution (*see* **A-8.4**),

V = total volume in ml of the clarified solution of the material prepared in **A-8.3**,

M = mass in g of the material taken for the test in **A-8.3**, and

A = percent moisture content of the sample as determined in **A-2**.

A-9. DETERMINATION OF WATER SOLUBLE SULPHATES

A-9.1 Apparatus

A-9.1.1 *Sintered Glass Crucible* (G-3)

A-9.1.2 *Hot Air Oven*

A-9.1.3 *Desiccator*

A-9.1.4 *Beakers* — 500-ml capacity.

A-9.2 Reagents

A-9.2.1 *Hydrochloric Acid* — concentrated.

A-9.2.2 *Barium Chloride* — 10 percent.

A-9.3 Procedure — Take 5 g of the sample in a 500-ml beaker, disslove in 100 ml of distilled water and filter through a Whatman filter paper No. 40. Wash the residue two to three times using about 10 ml of distilled water. Acidify the solution with about 10 ml of concentrated hydrochloric acid. Make up the solution with water to about 250 ml and boil. Add 5 ml of barium chloride solution to this boiling solution and allow to stand overnight. Filter the precipitate on a tared sintered glass crucible. Wash the residue with distilled water, dry and place in hot-air oven at 105 ± 2°C for 1 h. Cool the crucible to room temperature in a desiccator and weigh. Repeat till constant mass is obtained. Run a blank at the same time with all the reagents but without the sample.

A-9.4 Calculation

Water soluble sulphates, percent by mass

$$= \frac{(M_2 - M_1) \times 0{\cdot}746 \times 100}{M}$$

where

M_2 = mass of the crucible with residue,

M_1 = mass of the empty crucible, and

M = mass of the sample taken.

A-10. DETERMINATION OF WATER SOLUBLE CHLORIDES (VOLHARD'S METHOD)

A-10.1 Reagents

A-10.1.1 *Dilute Nitric Acid* — 1 : 4, free from lower oxides of nitrogen which give colour with thiocyanates.

A-10.1.2 *Standard Silver Nitrate Solution* — N/20.

A-10.1.3 *Standard Potassium Thiocyanate* — N/20.

A-10.1.4 *Ferric Alum Indicator* — 10 percent.

A-10.2 Procedure

A-10.2.1 Weigh accurately about 5 g of the sample and extract in about 100 ml of distilled water. Filter through a Whatman number 40 filter paper. Wash the residue using about 5 to 10 ml of distilled water. Add 20 ml of dilute nitric acid and a measured excess of silver nitrate solution (usually 20 ml). If a significant opalescence due to the precipitation of silver chloride is obtained, shake the flask well till all the silver chloride has coagulated and then titrate with potassium thiocyanate using 5 ml of ferric alum solution as internal indicator.

A-10.2.2 Run a blank using all the reagents but without the sample.

A-10.2.3 *Calculation*

Chlorides (as NaCl), percent by mass

$$= \frac{(V_1 - V_2) \times N \times F \times 0{\cdot}058\,46}{M} \times 100$$

where

V_1 = volume in ml of standard potassium thiocyanate required for the blank,

V_2 = volume in ml of standard potassium thiocyanate required for the sample,

N = normality of standard potassium thiocyanate solution,

F = factor of standard potassium thiocyanate, and

M = mass in g of the sample taken for test.

A-11. DETERMINATION FOR CARRYING OUT THE RETROGRADATION TEST

A-11.1 Procedure — Measure 100 ml of distilled water into a 400-ml beaker. Weigh 60 g of dextrin to the nearest 0·1 g and add it to the water in the beaker. Stir the mixture with a glass rod ensuring that none of the sample adheres to the side of the beaker. Boil the solution for one minute and set aside to cool overnight. Remove any crust that may have been formed and test the consistency of the paste by feel. The consistency should be that of lard. There should be no lumps and no free liquid.

A-12. PRACTICAL TEST

A-12.1 Procedure — A laboratory plant trial precipitation of lead azide with the sample dextrin should give round and uniform crystals.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF DEXTRIN FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

B-1. SCALE OF SAMPLING

B-1.1 Lot — In any consignment, all the bags of material of the same type and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-1.1.1 Tests for the determination of the conformity of a lot to the requirements of the specification shall be done for each lot separately. The number of bags to be selected at random for this purpose from a lot shall be as given below:

No. of Bags in the Lot	*No. of Bags to be Selected*
Up to 15	3
16 to 25	4
26 to 50	5
51 to 100	7
101 and above	10

B-2. PREPARATION OF TEST SAMPLES

B-2.1 From each of the bags selected according to **B-1.1.1**, small portions of the material shall be drawn with the help of a suitable sampling instrument from different parts of the bags. The total quantity of the material so drawn from each bag shall be not less than 200 g and these shall form the individual samples representative of the different bags selected.

B-2.2 From each of the individual samples formed according to **B-2.1**, approximately 150 g of the material shall be drawn and mixed together to form a composite sample. The composite sample so formed may be reduced further, if necessary, by coning and quartering so as to obtain enough material sufficient to conduct all the tests specified in this standard.

B-3. NUMBER OF TESTS

B-3.1 Tests for the determination of all characteristics mentioned in Table 1 shall be conducted on the composite sample (*see* **B-2.2**).

B-4. CRITERIA FOR CONFORMITY

B-4.1 The lot shall be declared as conforming to the requirements of the specification if the conditions stipulated in **B-4.1.1** are satisfied.

B-4.1.1 The test results for all the characteristics determined on the composite sample shall be found to be satisfactory.